AD/A-000 919

FIBER STRENGTHENED SUPERALLOYS

NUCLEAR METALS, INCORPORATED

PREPARED FOR

ARMY MATERIALS AND MECHANICS RESEARCH CENTER

JANUARY 1974

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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ABSTRACT

Discontinuous fibers of Mo were incorporated in matrices of nickel base superalloys INCO 625, INCO 713LC, IN-100, and TDNiCr by co-extrusion of blended powders. An elevated temperature stress rupture test showed that fiber strengthening was obtained only in INCO 625 in the temperature region of 1500 to 1700°F.

I. INTRODUCTION

The nickel superalloys are remarkable in that they retain their strengths up to a high fraction of their melting points. The conventional (cast and cast-and-wrought) superalloys derive their strength from the combination of solid solution hardening, the precipitation of a coherent gamma prime phase with the broad composition of Ni_3 [Al, Ti, Cb], and the presence of MC, $M_{23}C_6$ and M_6C carbides in the grains and at grain boundaries. The dispersion hardened alloys, which are always produced via PM processing, derive their properties from a uniform distribution of very fine, stable oxide particles, and (with the exception of TD nickel) solid solution strengthening. In earlier work sponsored by AMMRC(1), investigations have been performed into the feasibility of further property enhancement through the technique of in-situ refractory-metal fiber formation. This program is a continuation of that effort.

II. OBJECTIVE

The purpose of this program was to investigate the degree to which molybdenum primarily, and also niobium and titanium fibers produced in-situ by the extrusion of mixtures of matrix and fiber powders, could strengthen nickel superalloy matrices. The specific objective was to extrude the matrix-fiber combinations listed in Table I, to heat treat and form a favorable fiber-matrix reaction product, and then to stress-rupture test a selected number of samples.

III. TECHNICAL DISCUSSION

The matrix alloys under consideration were IN-100, 713LC, Inconel 625, nickel, and DS nickel chromium (DSNiCr); molybdenum, niobium and titanium were the fiber materials. The IN-100, 713LC and niobium were purchased in the form of cast bar, Inconel 625 and titanium as wrought bar, and the pure nickel and DSNiCr as powders, Table II. The bar alloys were centerless ground

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(1) Superalloy Matrix Refractory Metal Composites, DAAG46-71-C-0076 and 0148, P. Loewenstein, 1971.

TABLE 1. MATRIX-FIBER COMBINATIONS

1.	Inconel 625-35^{w}/o Mo	(1)
2.	Inconel 625-45^{w}/o Mo	(2)
3.	DSNiCr-35^{w}/o Mo	(2)
4.	DSNiCr-45^{w}/o Mo	(2)
5.	DSNiCr-30^{w}/o Nb	(1)
6a.	Ni-20^{w}/o Ti	(1)
6b.	DSNiCr-20^{w}/o Ti	(1)
7.	713LC-35^{w}/o Mo	(2)
8.	IN-100-35^{w}/o Mo	(2)
9.	IN-100-30^{w}/o Nb	(1)

TABLE II. MATRIX AND FIBER COMPOSITION

1.	CP Molybdenum:	99.98^{+}% Mo
2.	CP Niobium :	99.85^{+}% Nb; 0.073 Ta; 0.013 Ti; 0.01 Zr; 0.05 other
3.	CP Titanium :	99.47% Ti; 0.35 O; 0.14 Fe; 0.023 C; 0.011 N; 0.005 H
4.	Inconel 625 :	60.79% Ni; 22.27 Cr; 9.12 Mo; 3.55 Cb+Ta; 3.42 Fe; 0.26 Ti; 0.23 Al; 0.23 Si; 0.05 C; 0.05 Mn; 0.005 S
5.	DSNiCr :	78% Ni; 20.2 Cr; 1.7 ThO_2; 0.07 Fe; 0.01 Co; 0.02 excess O; 0.007 S; 0.004 C
6.	713LC :	74.2% Ni; 12.0 Cr; 6.17 Al; 0.76 Ti; 4.76 Mo; 1.96 Cb+Ta; 0.08 Co; 0.05 C; 0.006 S; 0.004 O
7.	IN-100 :	59.5% Ni; 15.4 Co; 10.5 Cr; 5.55 Al; 4.72 Ti; 3.02 Mo; 1.05 V; 0.16 C; 0.06 Zr; 0.015 B; 0.006 N; 0.005 O; 0.001 H

to 2.500 inches in diameter and converted to powder by the Rotating Electrode Process (REP). A description of this powder-making technique has been presented elsewhere[2]. The particle size distribution for the purchased powder and the REP powders is presented in Table III and Figures 1 and 2.

Five trial extrusions were performed in order to establish extrusion/fibering conditions for previously unexplored matrix-fiber combinations. Weighed amounts of powder (approximately 8-1/4 pounds for the molybdenum powder billets, and 5-3/4 pounds for the billets containing titanium) were mixed in a twin-cone blender and carefully transferred into steel extrusion cans to minimize powder segregation. The cans were welded, evacuated, outgassed to a pressure of 5×10^{-2} millitorr and sealed. The billets were heated on end and extruded under conditions deemed suitable for each of the powder combinations (Table IV).

The trial series established that NiCr/Mo, and IN-100/Mo could be extruded under the given conditions. The 713LC/Mo was too stiff under trial conditions (a pressure of 150 tons/inch^2 places too high a stress on our tooling to be allowed on a scheduled basis). The NiCr-Ti combination extruded at 1715°F was also too stiff to be relied upon as a repeatable extrusion condition.

Metallographic examination of longitudinal sections taken from the center of each bar showed the NiCr/Mo, 713LC/Mo and IN-100/Mo combinations all possessed satisfactory arrays of molybdenum fibers, while neither NiCr/Ti extrusions showed any trace of the admixed titanium powders. It is assumed that the titanium was completely consumed in liquid/solid reactions. Based on these observations, the following decisions were made:

1. NiCr/Mo and IN-100/Mo would be extruded as in the trials.
2. Extrusion temperature for 713LC/Mo would be raised 25°F, and the reduction ratio dropped slightly (to 9.8:1).
3. The combination NiCr/Ti would be replaced by Ni/Ti, using INCO Type 123 carbonyl nickel and REP titanium. The Ni/Ti would be extruded through a 11:1 reduction at 1600°F.

The balance of the refractory metal-superalloy billets were then extruded under the conditions indicated in Table V. Four longitudinal samples, representing the front, first third, second third and rear of each matrix-fiber

TABLE III. MATRIX AND FIBER PARTICLE SIZE DISTRIBUTION

		Fraction Retained on Screen, %								
U. S. Screen Size:		35	45	60	80	120	170	230	325	PAN
Microns:		500	354	250	177	125	88	63	44	44
1.	Mo :	.04	.4	8.31	41.21	33.34	12.11	3.35	1.03	.21
2.	Nb :	.01	.17	4.31	27.19	42.83	16.83	6.11	1.95	.60
3.	Ti :	.04	6.21	27.39	37.36	18.69	6.49	2.81	.90	.12
4.	Inconel 625 :	.05	.14	3.76	19.70	36.02	19.88	13.19	7.22	.04
					150					
5.	NiCr :	0	0	0	.03	-----	-----	6.8	21.00	71.9
6.	713LC :	0	10.58	44.72	28.88	10.39	3.82	1.19	.37	.05
7.	IN-100 :	0	0	0	19.47	39.14	20.80	14.18	6.41	0

Figure 1. Particle Size Distribution, Matrix Powders

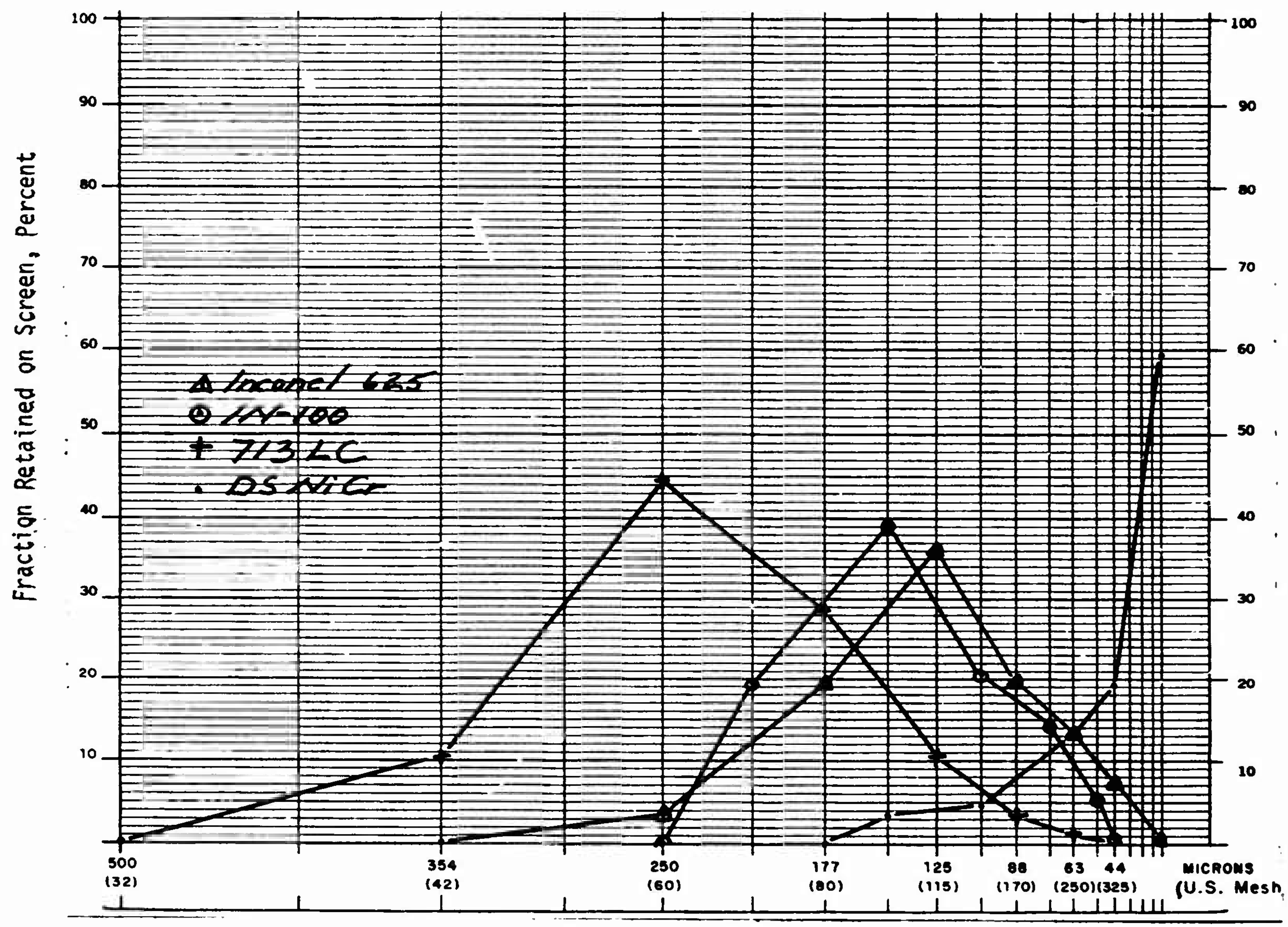


Figure 2. Particle Size Distribution, Fiber Powders

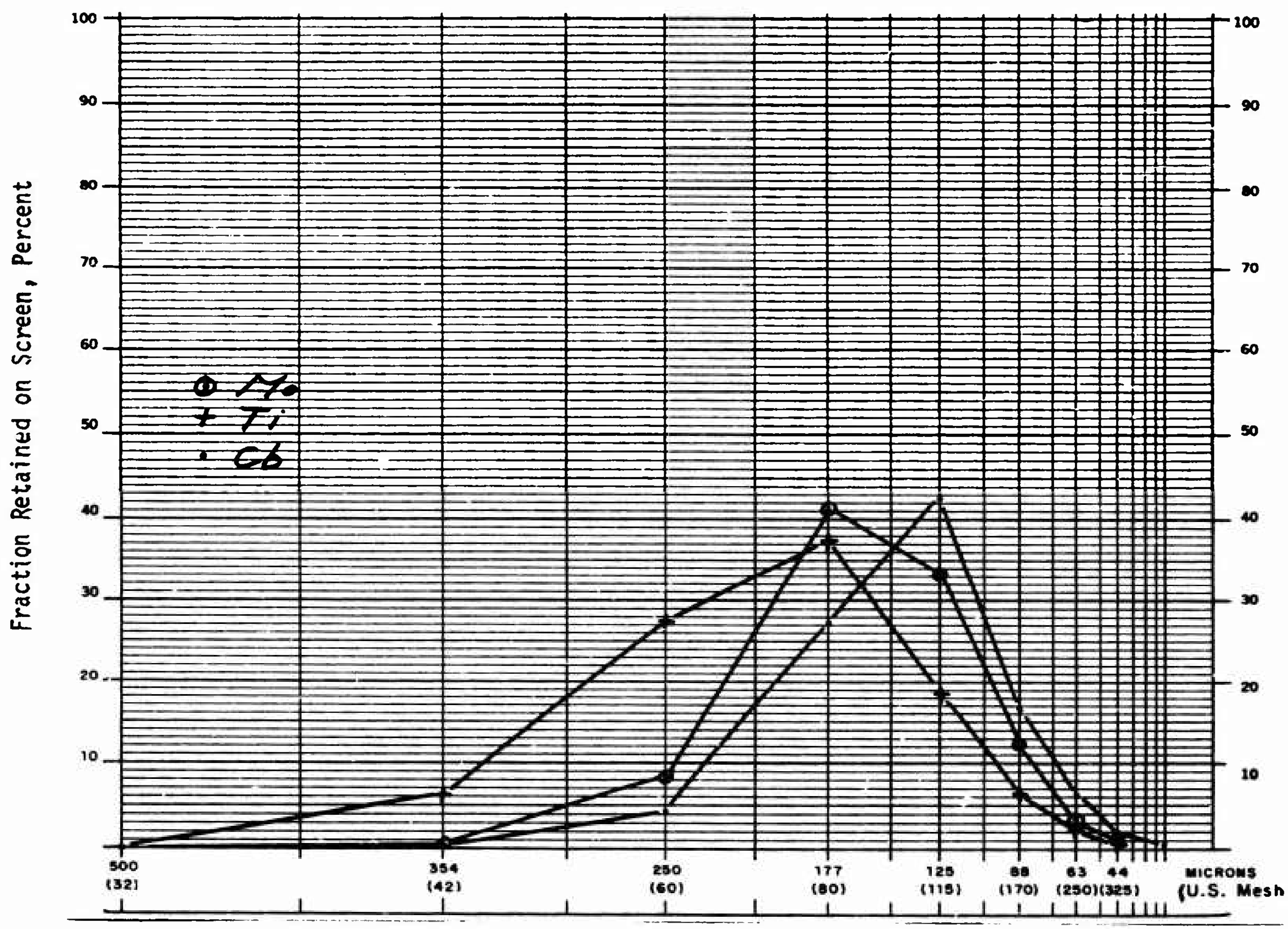


TABLE IV

TRIAL EXTRUSION OF FIVE REFRACTORY METAL SUPERALLOY BILLETS

Billet Diameter: 3" Die Size: 0.940" Reduction Ratio: 11:1

No.	Composition w/o	Temp. (°F)	UPSET			RUN		
			Force (ton)	Pressure (tsi)	K (tsi)	Force (ton)	Pressure (tsi)	K (tsi)
1	55%NiCr-45%Mo	1950	640	83.4	34.7	550	71.7	22.9
2	65%713LC-35%Mo	1950	805	105	43.7	805	105	43.7
3	80%NiCr-20%Ti	1715	800	104	43.3	800	104	43.3
4	65%IN-100-35%Mo	2000	690	90.0	37.4	610	79.5	33.1
5	80%NiCr-20%Ti	1755	730	95.2	39.6	755	98.4	41.0

TABLE V

EXTRUSION OF REFRACTORY METAL-SUPERALLOY POWDER BILLETS

Billet Diameter: 3"   Die Size: 0.940"   Reduction Ratio: 11.1

Composition	Temp. (°F)	UPSET			RUN		
		Force (ton)	Pressure (tsi)	K (tsi)	Force (ton)	Pressure (tsi)	K (tsi)
80%Ni-20%Ti	1600	460	60	25	400	52.1	21.7
65%Inconel 625-35%Mo	1900	700	91.3	38.0	650	84.7	35.2
55%Inconel 625-45%Mo	1900	735	95.8	39.9	660	86.0	35.8
Ditto	1900	665	86.7	27.8	615	80.2	33.4
70%NiCr-30%Nb	1925	570	74.3	30.9	500	65.2	27.1
65%NiCr-35%Mo	1950	530	69.1	28.8	500	65.2	27.1
Ditto	1950	515	67.1	27.9	480	62.6	26.1
55%NiCr-45%Mo	1950	580	75.6	315	525	68.4	28.5
65%713LC-35%Mo*	1975	700	91.2	40.0	645	84.1	36.9
65%IN-100-35%Mo	2000	700	91.2	40.0	650	84.7	35.3
70%IN-100-30%Nb	2000	600	78.2	325	530	69.1	28.8

* 1" diameter die, R = 9.8:1

combination were then prepared for metallographic examination (Figure 3), with the balance of the extrusions submitted to AMMRC for heat treatment.

Seventy (70) samples were returned by AMMRC and were machined into standard ASTM 0.250-inch diameter stress-rupture test bars. Rupture tests were performed in air, with specimens soaked two to three hours at temperature prior to load application. Test parameters and results are tabulated in Table VI.

The time, temperature and rupture life for each specimen was combined in a single parametric term which could be plotted against rupture stress. The Larson-Miller parameter

$$P = T(C + \text{Log } t) \times 10^{-3}$$

T = Temperature, °Rankine

C = Constant, assumed to be 20

t - rupture life, hours

was then calculated for each test specimen and the results superimposed on a Larsen-Miller plot for each matrix alloy computed from INCO data (Table VII and Figures 4, 5, 6, and 7).

The figures clearly show the relationship between the rupture strengths of the matrix alloys, the pure fiber metals, and the fiber-reinforced matrices. It can be seen that the Inconel 625-Mo composites are the only ones in which fiber-strengthening has occurred; this is the only fiber-matrix pair in which there exists a temperature region in which the fiber metal is intrinsically stronger than the matrix alloy.

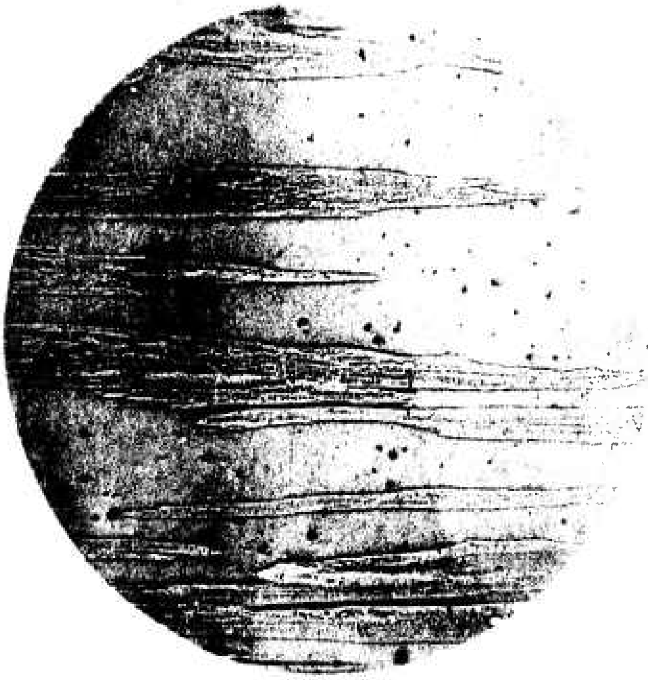
The heat treatment of the extruded bars reacted the fibers with the matrices, Figure 8, in the hope that the Mo-Ni-Cr intermetallic zone thus formed would cause significant matrix strengthening. This appears not to be the case since, as noted above, the only property-improvement achieved was that to be expected from the separate strengths of molybdenum and Inconel 625.

The rupture testing of these specimens revealed another area of concern with respect to the use of refractory-metal fibers in air.

Figure 9 illustrates the loss by oxidation of the refractory-metal fibers protruding through the threads and at the end face of a rupture specimen. The molybdenum has reacted with the air to form MoO_3, a volatile specie. The photomicrographs of Figure 10 show the fracture paths of the ends of test specimens, and further illustrate the relative weakness and low ductility of the molybdenum fibers.

Figure 3. Fiber Strengthened Superalloys.
Longitudinal Sections. 75X

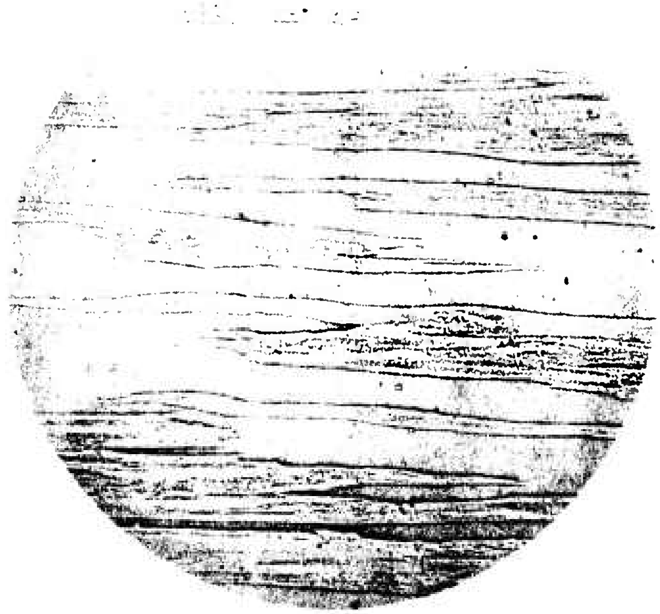
1. Inconel 625-35^{W}/oMo



FRONT

FRONT-MIDDLE

MIDDLE-REAR



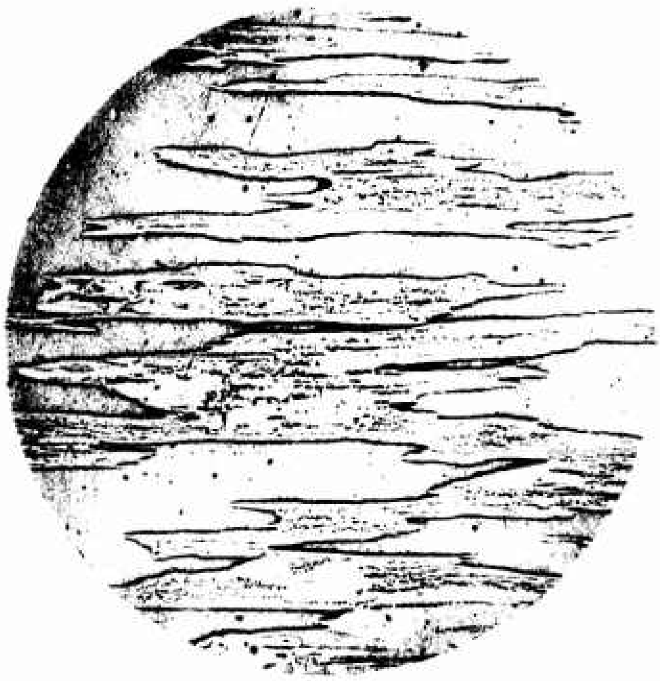
REAR

Figure 3. (Continued)

2. Inconel 625-45^{W}/oMo

FRONT

FRONT-MIDDLE



MIDDLE-REAR

REAR

Figure 3. (Continued)

3. DSNiCr-35^{W}/oMo

FRONT

FRONT-MIDDLE

MIDDLE-REAR

REAR

Figure 3. (Continued)

4. DSNiCr-45^{W}/oMo

FRONT

FRONT-MIDDLE

MIDDLE-REAR

REAR

Figure 3. (Continued)

5. DSNiCr-30^{W}/oNb

FRONT

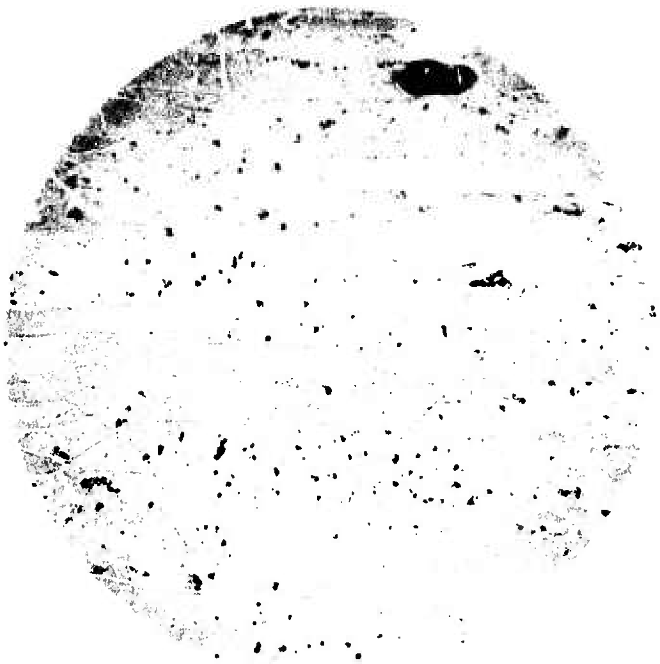
FRONT-MIDDLE

MIDDLE-REAR

REAR

Figure 3. (Continued)

6. DSNiCr-20^{W}/oTi



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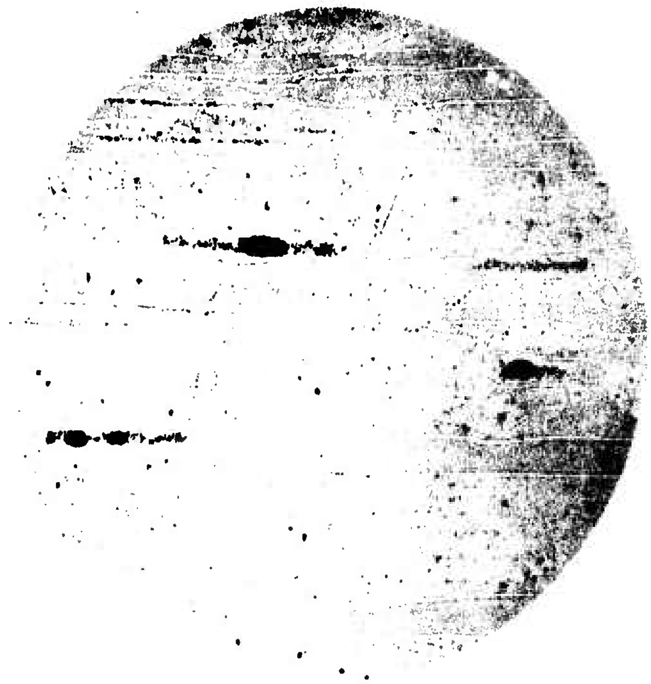
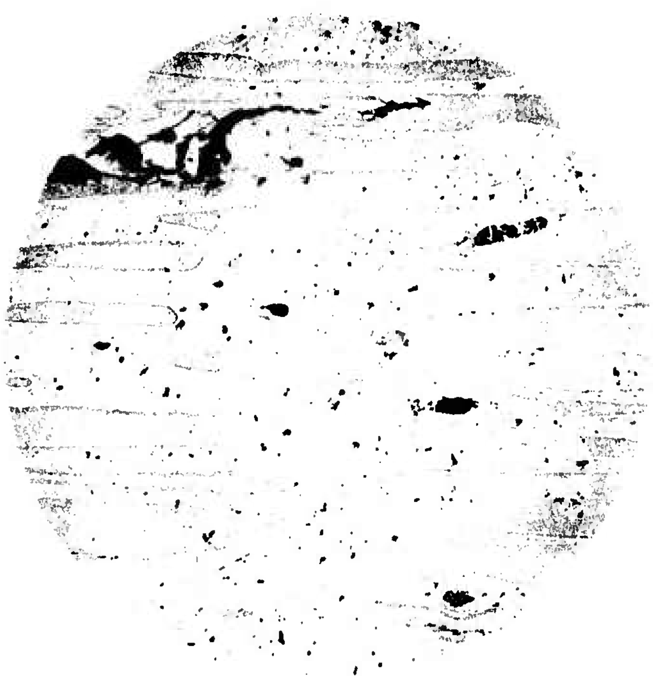
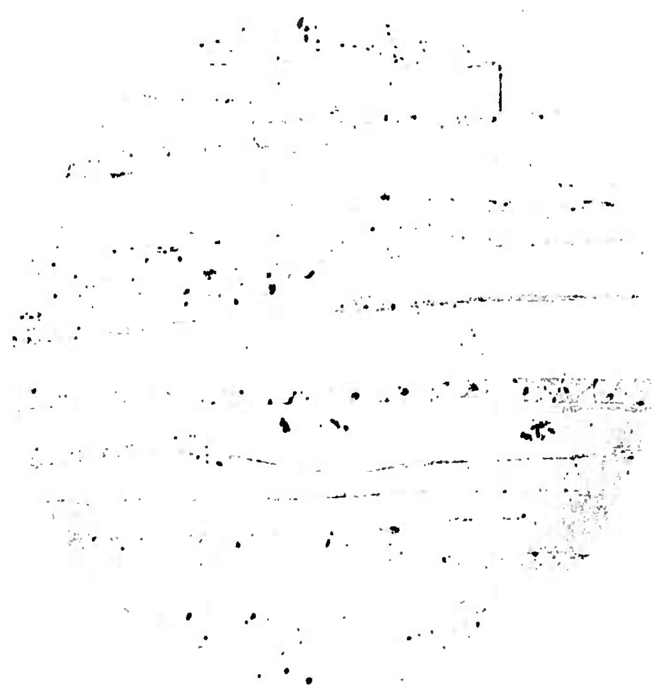


FRONT-MIDDLE



MIDDLE-REAR

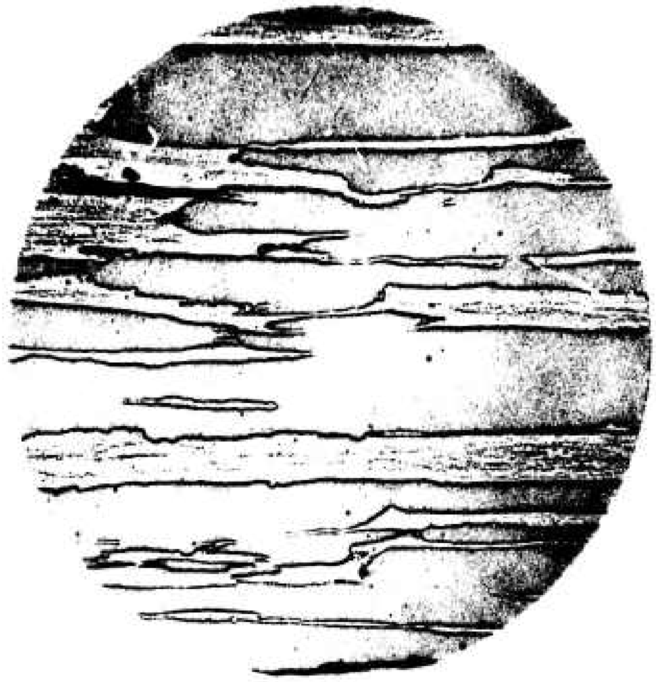


REAR

Figure 3. (Continued)

7. 713LC-35 w/o Mo

FRONT



FRONT-MIDDLE

MIDDLE-REAR

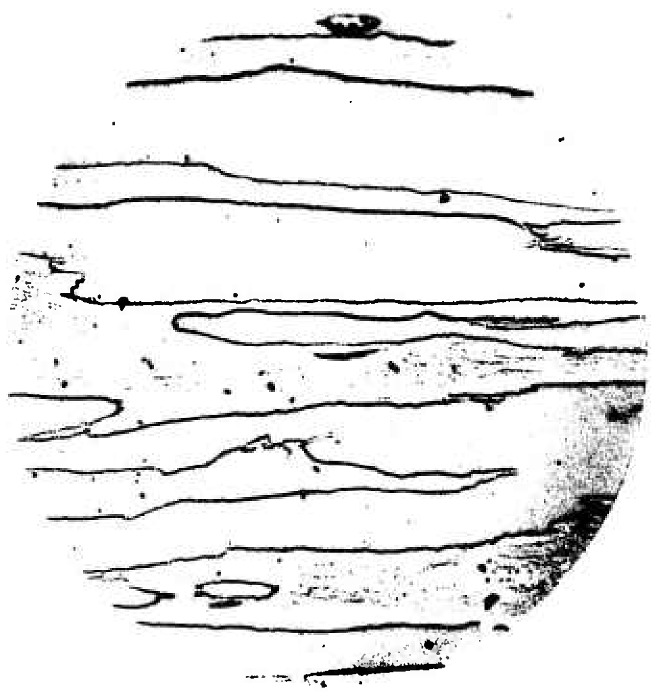


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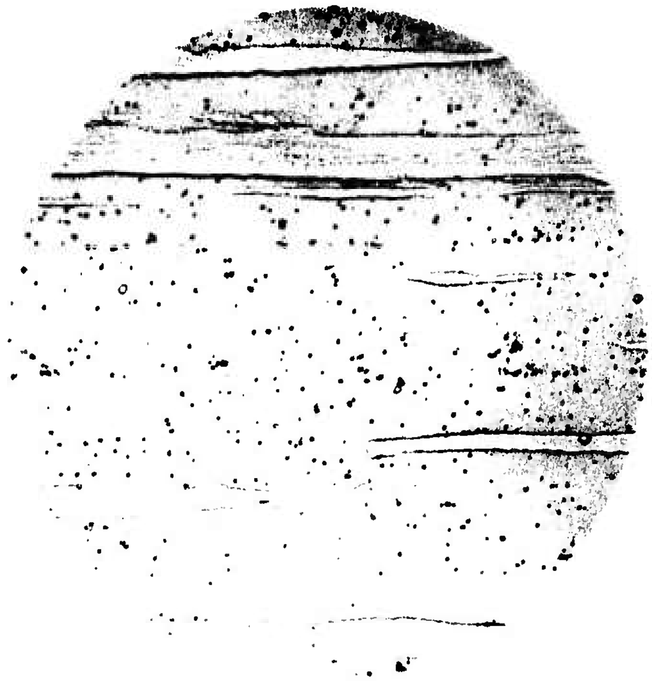
Figure 3. (Continued)

8. IN-100-35^{W}/oMo

FRONT



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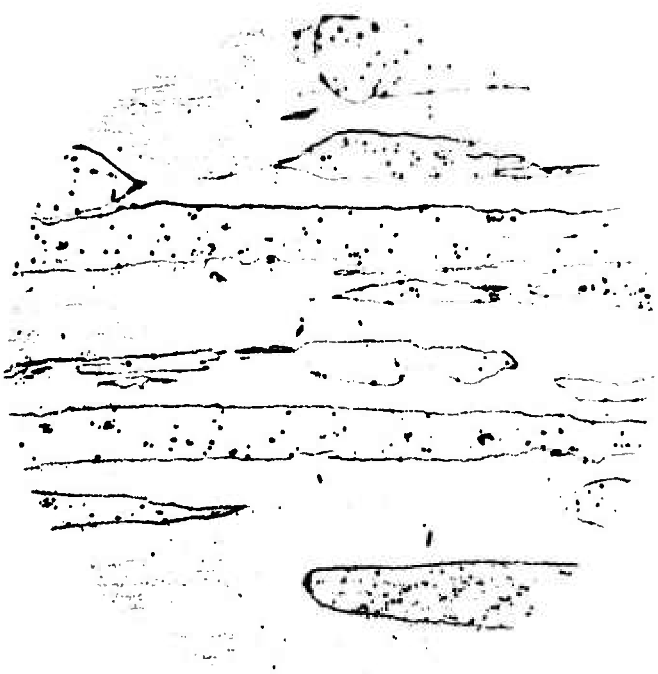


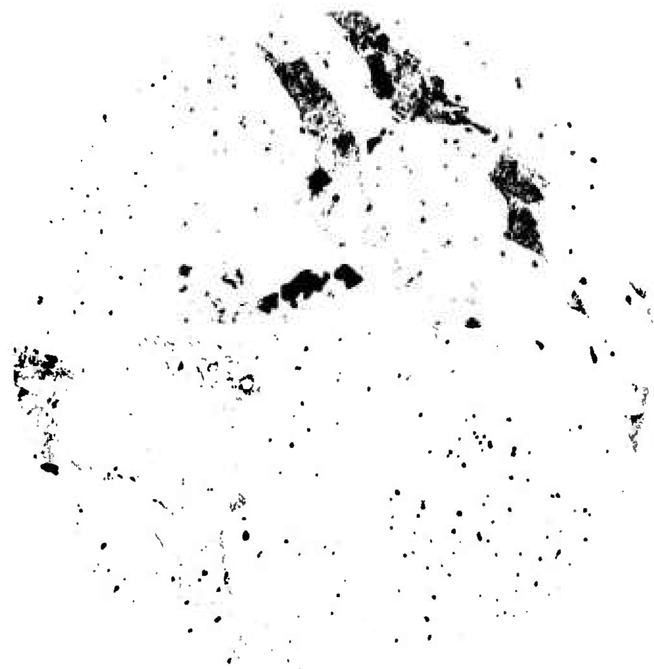
MIDDLE-REAR

REAR

Figure 3. (Continued)

9. IN-100-30^{W}/oNb

FRONT



FRONT-MIDDLE

MIDDLE-REAR

REAR

TABLE VI. STRESS-RUPTURE LIFE FOR FIBER-STRENGTHENED SUPERALLOYS

MATRIX/FIBER	SAMPLE NO.	TEMP. (°F)	STRESS (KSI)	LIFE (HRS)	% ELONG. (1" G.L.)	% R.A.
Inconel 625-35 W/oMo						
	A1	1500	30	5.2	50.3	55.4
	A5	1500	30	4.0	60.1	51.5
	A2	1500	25	8.4	46.8	51.5
	A6	1500	20	42.7	55.8	54.7
	A9	1700	20	3.2	16.5	20.5
	A10	1700	10	47.3	23.2	23.8
Inconel 625-45 W/oMo						
	B1	1500	35	2.8	74.4	56.6
	B4	1500	35	1.6	49.4	----
	B2	1500	25	11.0	66.8	54.0
	B5	1500	25	14.8	54.3	45.8
	B8	1700	20	3.6	11.4	19.3
	B9	1700	10	6.2	----	17.2
	B10	1750	20	1.4	14.8	14.3
	B11	1750	10	19.6	----	20.6
DSNiCr-35 W/oMo						
	K1	1700	20	0.1	11.3	15.1
	K5	1700	20	0.3	2.6	4.7
	K2	1700	10	6.1	10.4	15.2
	K6	1700	10	7.3	5.6	7.7
	K7	1800	20	0.1	4.5	5.8
	K8	1800	10	1.2	5.6	10.2
	K9	1900	20	0.1	7.2	5.6
	K10	1900	10	0.1	7.7	11.3
	L11	2000	10	0.1	5.7	7.3
	L12	2000	10	0.1	5.4	8.0
DSNiCr-45 W/oMo						
	J1	1700	20	0.1	5.3	12.5
	J3	1700	20	1.3	3.4	5.6
	J2	1700	10	2.4	4.6	9.0
	J4	1700	10	25.8	7.3	12.4
	J5	1800	20	0.2	2.1	5.5
	J6	1800	10	2.2	4.2	9.8
	J7	1900	20	0.1	2.6	4.9
	J8	1900	10	0.4	4.7	9.6
	J9	2000	20	0.1	3.7	5.0
	J10	2000	10	0.1	10.0	8.7
DSNiCr-30 W/oNb						
	M1	1700	20	0.1	----	6.7
	M9	1700	20	0.1	3.6	4.2
	M2	1700	15	0.2	8.9	11.4
	M10	1700	15	0.4	2.1	3.9
Ni-20 W/oTi						
	N1	1500	45	0.1	8.8	----

TABLE VI. (Continued)

MATRIX/FIBER	SAMPLE NO.	TEMP. (°F)	STRESS (KSI)	LIFE (HRS)	% ELONG. (1" G.L.)	% R.A.
713LC-35^{W}/oMo						
	D2	1600	50	0.5	7.0	----
	E1	1600	50	0.2	9.2	----
	D3	1600	40	2.1	9.0	----
	E2	1600	40	0.7	7.2	----
	D4	1700	40	0.2	6.1	10.3
	D5	1700	30	0.8	8.5	9.6
	E5	1800	25	0.4	2.7	6.0
	E6	1800	15	3.8	3.6	5.9
IN-100-35^{W}/oMo						
	F1	1600	60	0.1	9.7	12.4
	F3	1600	60	0.1	7.1	8.9
	F2	1600	50	0.6	7.9	12.8
	F4	1600	50	0.4	10.4	9.8
	F5	1700	45	0.1	4.9	12.4
	F6	1700	30	0.2	----	24.4
	F7	1800	30	0.1	----	----
	F8	1800	20	0.3	----	7.9
	F9	1900	20	0.2	7.7	11.0
	F10	1900	15	0.2	16.8	28.4
IN-100-30^{W}/oNb						
	P6	1700	45	0.1	----	8.6

TABLE VII. LARSEN-MILLER PARAMETERS

$$P = T\ (C + \mathrm{Log}t) \times 10^{-3}$$
$$P = T\ (20 + \mathrm{Log}t) \times 10^{-3}$$

MATRIX/FIBER	SAMPLE NO.	TEMP. (°F)	TIME (HRS)	P	STRESS (KSI)
Inconel 625-35^{W}/oMo					
	A1	1500	5.2	40.6	30
	A2	1500	8.4	41.0	25
	A5	1500	4.0	40.4	30
	A6	1500	42.7	42.4	20
	A9	1700	3.2	44.3	20
	A10	1700	47.3	46.8	10
Inconel 625-45^{W}/oMo					
	B1	1500	2.8	40.1	35
	B2	1500	11.0	41.2	25
	B4	1500	1.6	39.6	35
	B5	1500	14.8	41.5	25
	B8	1700	3.6	44.4	20
	B9	1700	6.2	44.9	10
	B10	1750	1.4	44.5	20
	B11	1750	19.6	47.0	10
DSNiCr-35^{W}/oMo					
	K1	1700	0.1	41.0	20
	K2	1700	6.1	44.9	10
	K5	1700	0.3	42.1	20
	K6	1700	7.3	45.1	10
	K7	1800	0.1	42.9	20
	K8	1800	1.2	45.4	10
	K9	1900	0.1	44.8	20
	K10	1900	0.1	44.8	10
	L11	2000	0.1	46.7	10
	L12	2000	0.1	46.7	10
DSNiCr-45^{W}/oMo					
	J1	1700	0.1	41.0	20
	J2	1700	2.4	44.0	10
	J3	1700	1.3	43.4	20
	J4	1700	25.8	46.2	10
	J5	1800	0.2	43.6	20
	J6	1800	2.2	46.0	10
	J7	1900	0.1	44.8	20
	J8	1900	0.4	46.3	10
	J9	2000	0.1	46.7	20
	J10	2000	0.1	46.7	10
DSNiCr-30^{W}/oNb					
	M1	1700	0.1	41.0	20
	M2	1700	0.2	41.7	15
	M9	1700	0.1	41.0	20
	M10	1700	0.4	42.3	15

TABLE VII. (Continued)

MATRIX/FIBER	SAMPLE NO.	TEMP. (°F)	TIME (HRS)	P	STRESS (KSI)
Ni-20^{W}/oTi	N1	1500	0.1	37.2	45
713LC-35^{W}/oMo	D2	1600	0.5	40.5	50
	D3	1600	2.1	41.9	40
	D4	1700	0.2	41.7	40
	D5	1700	0.8	43.0	30
	E1	1600	0.2	39.8	50
	E2	1600	0.7	40.9	40
	E5	1800	0.4	44.3	25
	E6	1800	3.8	46.5	15
IN-100-35^{W}/oMo	F1	1600	0.1	39.1	60
	F2	1600	0.6	40.7	50
	F3	1600	0.1	39.1	60
	F4	1600	0.4	40.4	50
	F5	1700	0.1	41.0	45
	F6	1700	0.2	41.7	30
	F7	1800	0.1	42.9	30
	F8	1800	0.3	44.0	20
	F9	1900	0.2	45.6	20
	F10	1900	0.2	45.6	15
IN-100-30^{W}/oNb	P6	1700	0.1	41.0	45

FIGURE 4.

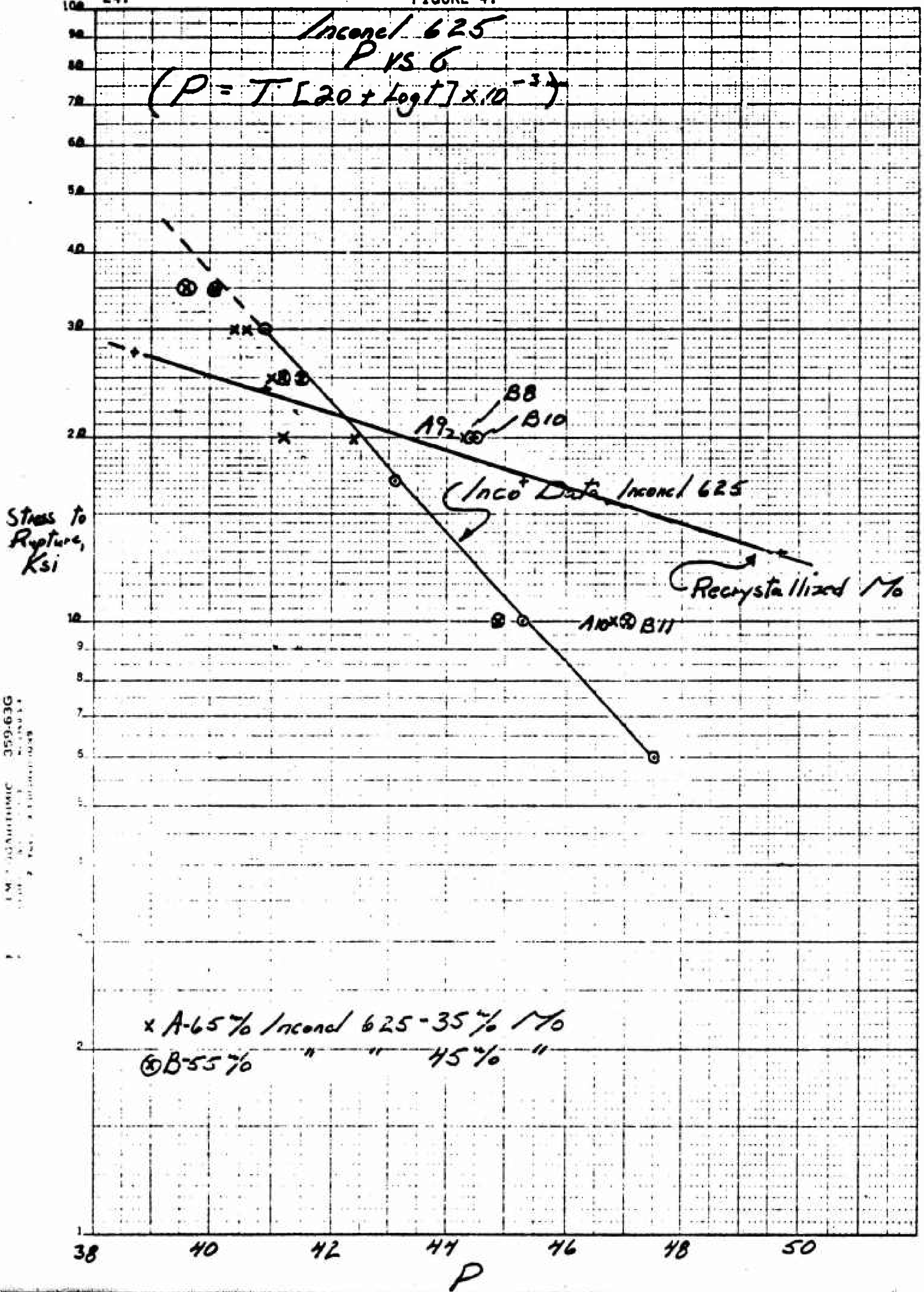

Inconel 625
P vs σ
(P = T [20 + Log t] x 10^-3)
Stress to Rupture, Ksi
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
2
1
B8
A9
B10
Inco Data Inconel 625
Recrystallized Mo
A10
B11
x A-65% Inconel 625-35% Mo
⊗ B-55% " " 45% "
38
40
42
44
46
48
50
P
359-63G

FIGURE 5.

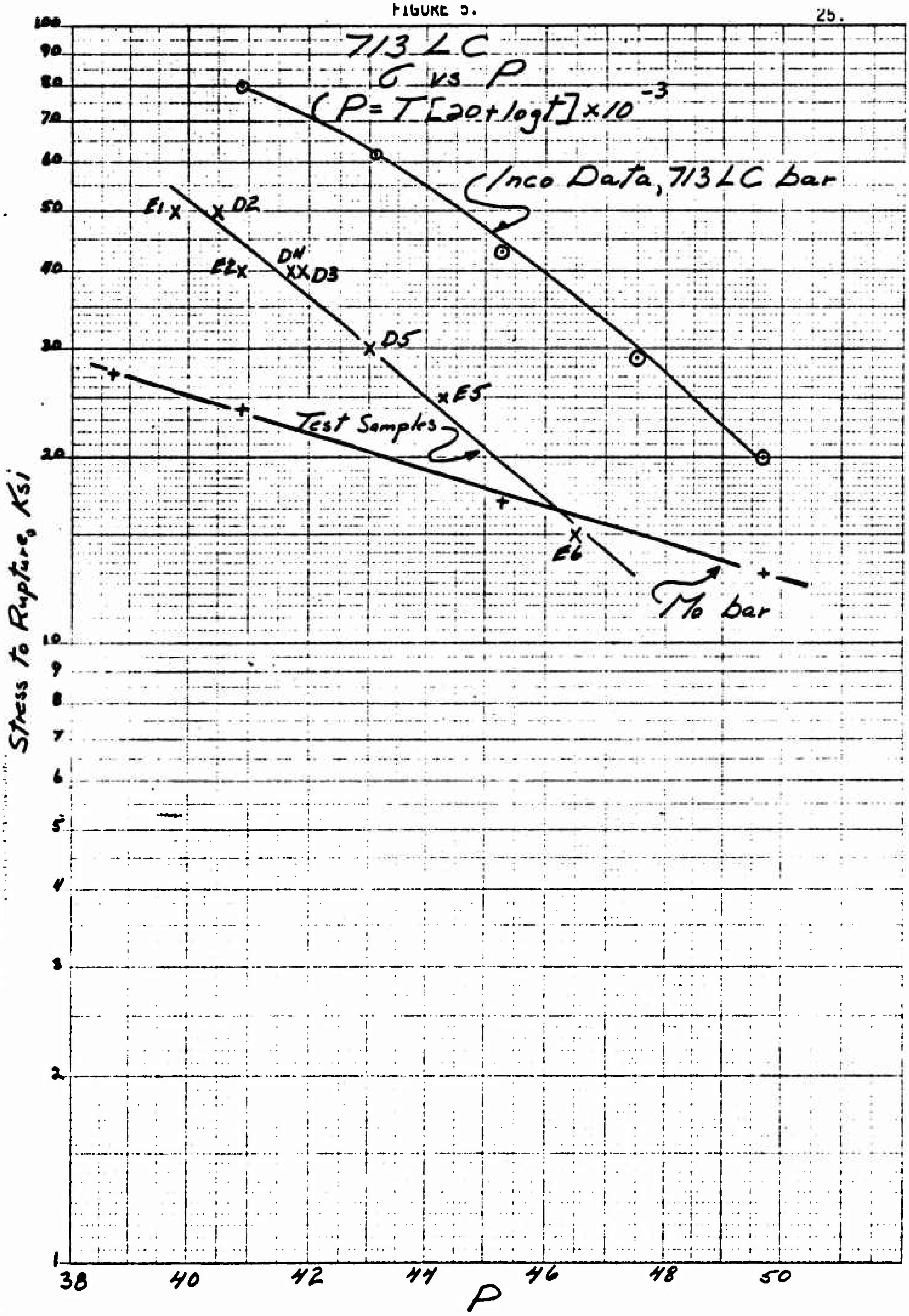
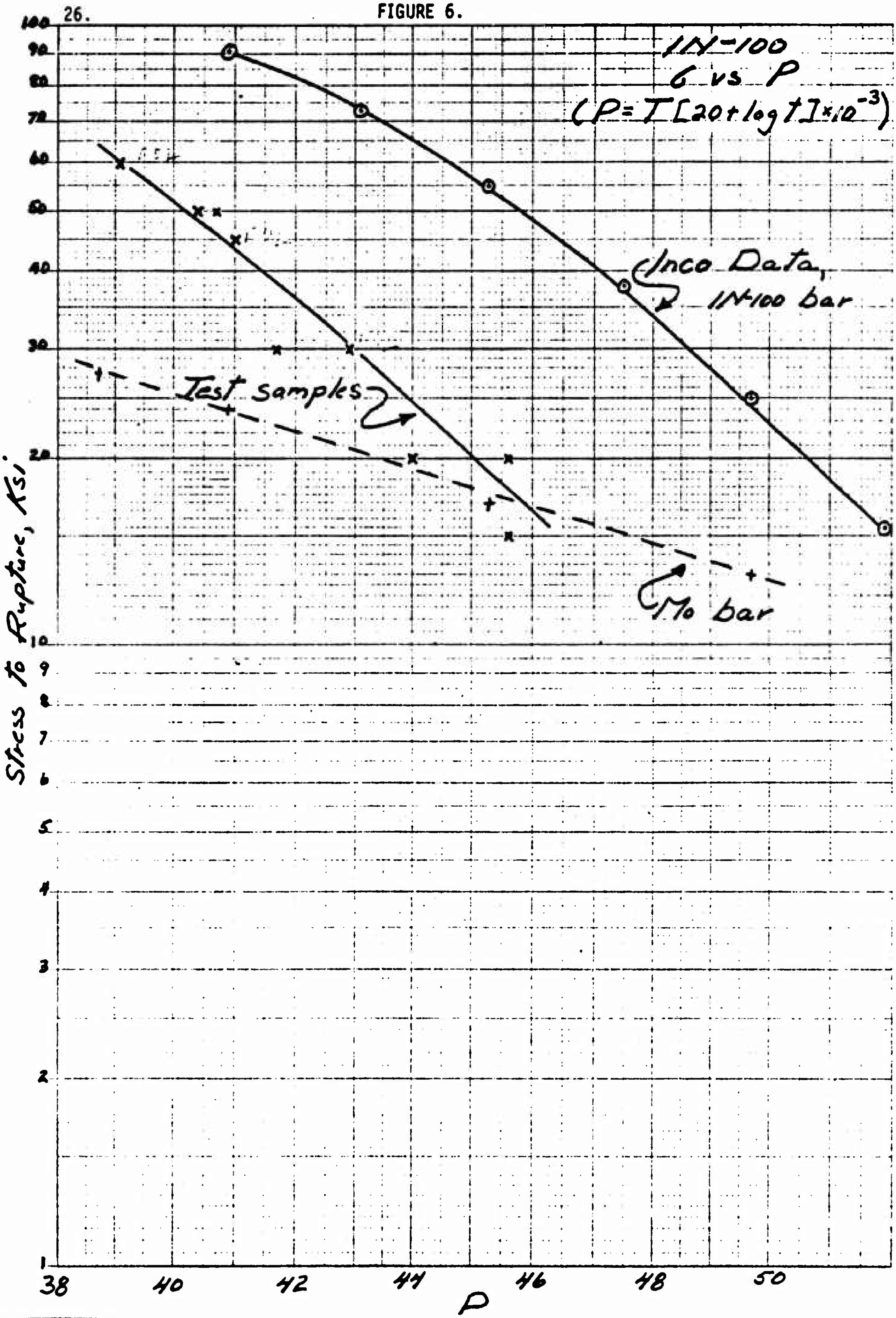
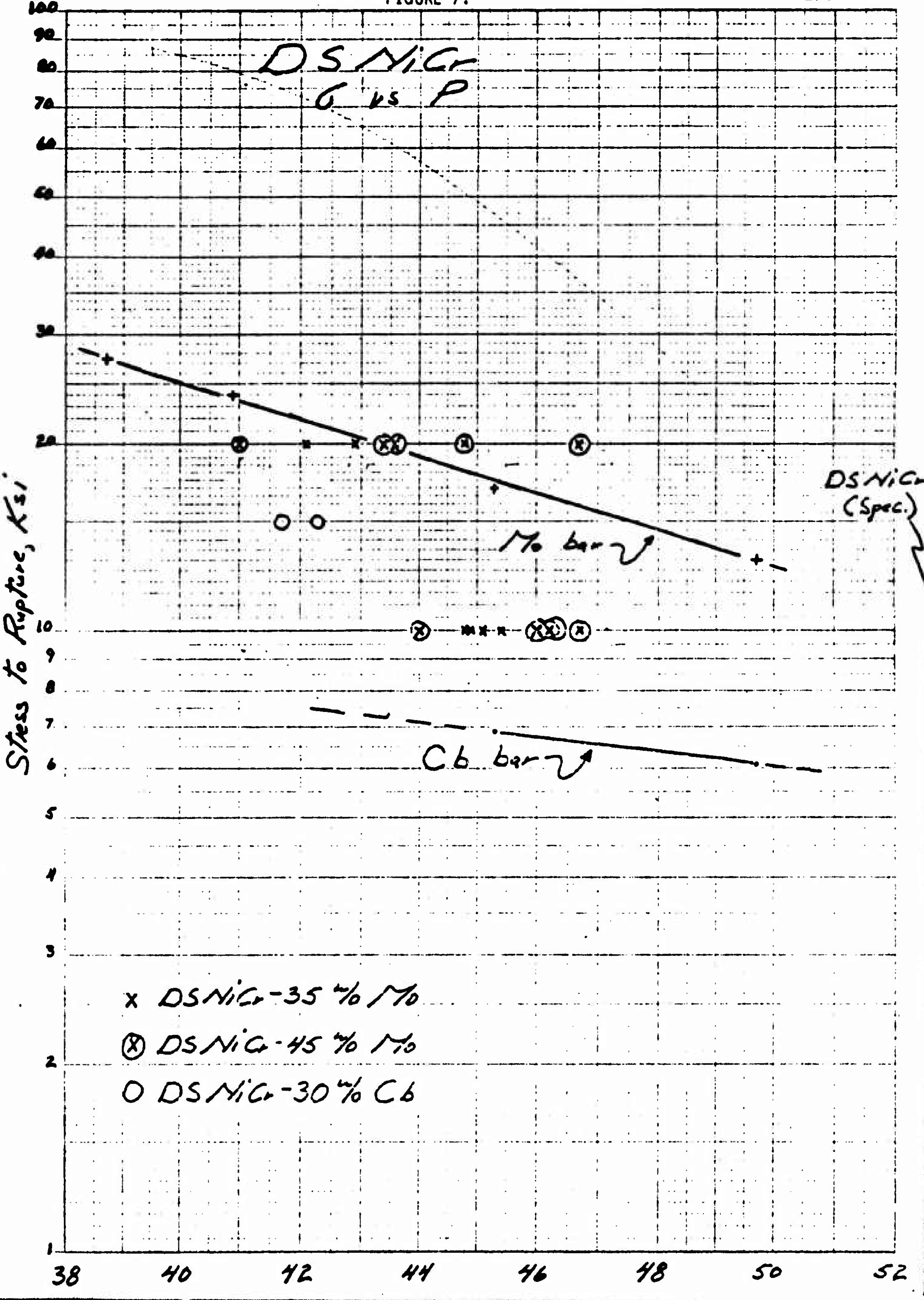

713 LC
σ vs P
(P=T[20+log t]×10⁻³
Inco Data, 713 LC bar
E1 x
D2
E2 x
D4
D3
D5
E5
Test Samples
E6
Mo bar
Stress to Rupture, Ksi
P
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2
1
38
40
42
44
46
48
50

FIGURE 6.



IN-100
σ vs P
(P = T[20 + log t] × 10^-3)
Inco Data, IN-100 bar
Test samples
Mo bar
Stress to Rupture, Ksi
P
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2
1
38
40
42
44
46
48
50

FIGURE 7.


DS NiCr
σ vs P
Stress to Rupture, Ksi
DS NiCr (Spec.)
Mo bar
Cb bar
x DS NiCr-35 % Mo
⊗ DS NiCr-45 % Mo
O DS NiCr-30 % Cb
100
90
80
70
60
50
40
30
20
10
9
8
7
6
5
4
3
2
1
38
40
42
44
46
48
50
52

A. Inconel 625-Mo (A10)

B. DSNiCr-Mo (K7)

Figure 8. Matrix-Fiber Reaction. 150X

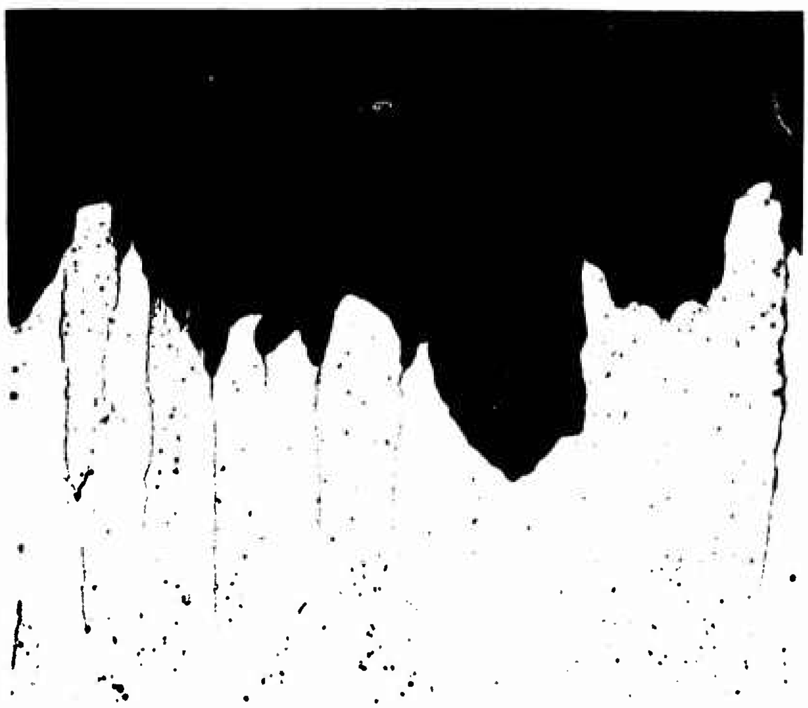


Figure 9. Fiber Oxidation of Specimen Ends,
IN-100-Mo (F1) 75X

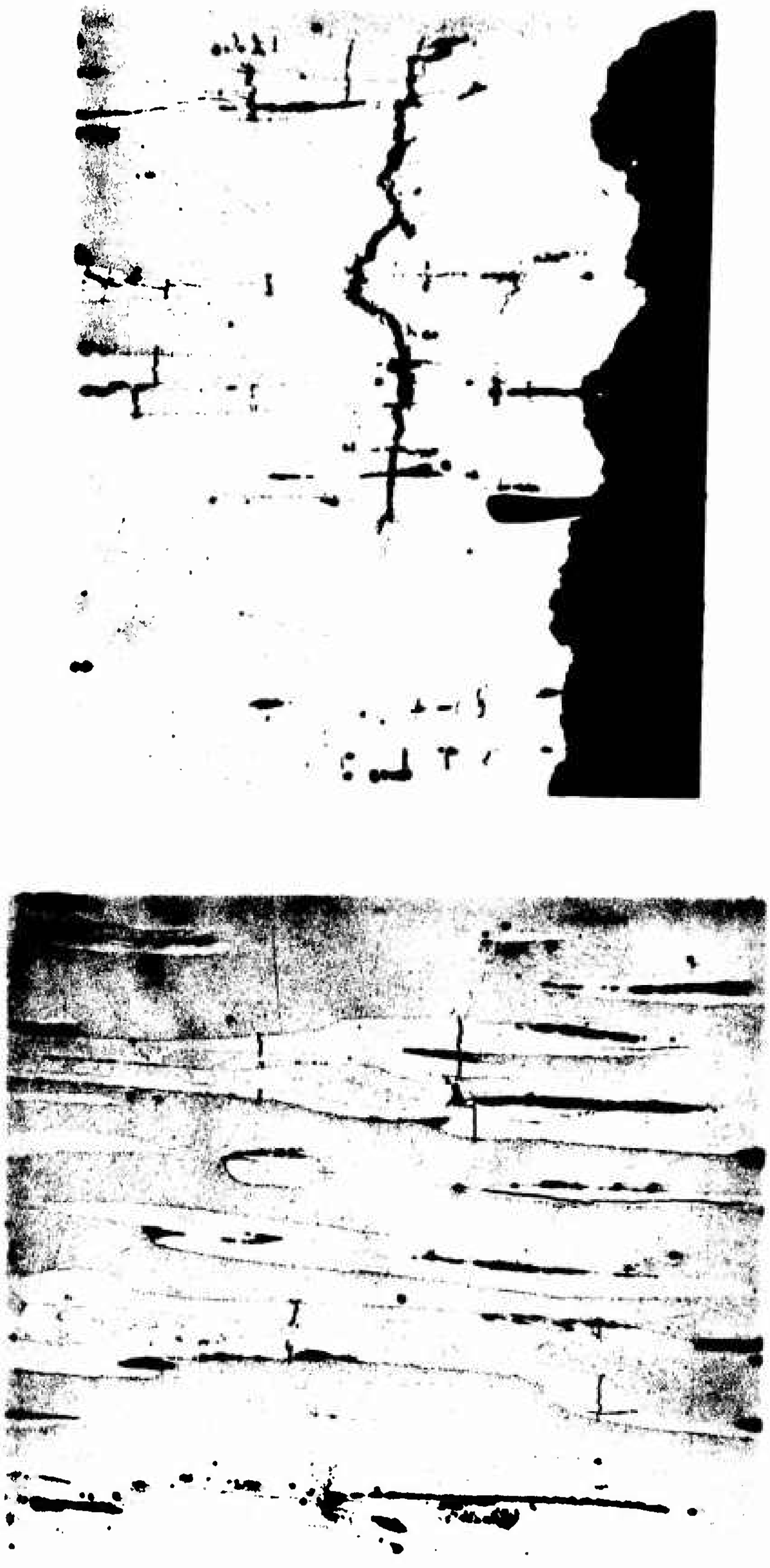


Figure 10. Fracture Path in DSNiCr (K7) 75X

IV. RECOMMENDATIONS

1. It would be useful to evaluate W-Re-Hf-C, which is significantly stronger than pure molybdenum, as a PM fiber strengthener for nickel superalloys.

2. A PM modification of the appropriate solution treatment and age cycle for each superalloy should be used for the fiber-matrix diffusion heat treatment.

3. A superalloy matrix containing W-Re-Hf-C fibers should be rupture-tested in air with and without an oxidation-resistant coating to determine the effect of surface oxidation of the fibers on sample ductility.